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Progress Report No. 820-27

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# INVESTIGATION OF LIQUID ROCKET PROPELLANTS

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**Contract N7onr-462**

### Task Order No. III

**Project No. NR 220 023**

**APR 15 1952**

**Aerojet**

A DIVISION OF

# THE GENERAL TIRE & RUBBER COMPANY

**THE  
GENERAL  
TIRE**

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24 March 1952

Progress Report No. 820-27

## INVESTIGATION OF LIQUID ROCKET PROPELLANTS

Contract N7onr-462  
Task Order III  
Project No. NR 220 023

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AEROJET ENGINEERING CORPORATION

Azusa, California

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## CONTRACT FULFILLMENT STATEMENT

This is the twenty-seventh in a series of progress reports submitted in partial fulfillment of Contract N7onr-462, Task Order III, covering the work performed in January and February 1952.\*

\* Work performed during the months of November and December 1951 has been included in Aerojet Report No. 577, the annual report on this contract.

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DEFINITION OF SYMBOLS

<u>Symbol</u>	<u>Description</u>	<u>Units</u>
$C_F$	Nozzle Coefficient	---
$A_t$	Nozzle Throat Area	in. <sup>2</sup>
$A_e$	Nozzle Exit Area	in. <sup>2</sup>
$c^*$	Characteristic Exhaust Velocity	ft/sec
$I_{sp}$	Specific Impulse	lb-sec/lb
$F$	Thrust	lb
$p_c$	Chamber Pressure	lb/in. <sup>2</sup>
$D_t$	Nozzle Throat Diameter	in.
$L^*$	Characteristic Chamber Length	in.



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## I. INTRODUCTION

A. Attention has been divided approximately equally among the three distinct phases of the contract during this report period: research on the development or improvement of rocket propellants; design and operation of a pilot plant for the production of commercially unobtainable propellants; and evaluation of experimental performance of new propellants in a small rocket engine.

B. Laboratory investigations were concerned with research on the methyl derivatives of hydrazine, and the kinetics of the thermal decomposition of nitromethane under high pressure. The desired goal of the investigation of derivatives of hydrazine is the development of a fuel possessing the performance of hydrazine, but with substantially improved physical properties. The aim of the study of nitromethane is to extend the knowledge of the mechanism of the thermal decomposition of the monopropellant to include pressures more comparable to actual rocket-motor conditions than those used by previous workers.

C. The preparation of approximately 40 lb of a 13.2% solution of lithium borohydride in hydrazine represents the present pilot-plant program; several minor modifications in the previously reported procedure were made after the first preliminary run, and it appears that both high purity and high yields will be obtained in present production runs.

D. Although no extensive single program has been conducted with the propellant test engine during this period, an interesting series of tests was made with an oxidizer consisting of a mixture of oxygen and fluorine. The engine thrust was increased from 50 to 100 lb to improve experimental accuracy. The performance of hydrazine burned with oxygen was studied both to establish the operating characteristics of the modified engine, and to determine a base curve for comparison with the one to be obtained with the low-freezing solution of lithium borohydride.

## II. STUDY OF HYDRAZINE DERIVATIVES

### A. INTRODUCTION

1. The commercial availability of 96% hydrazine in recent years, and the incentive provided by the excellent performance and burning characteristics of hydrazine when used as a rocket fuel, have resulted in several extensive programs devoted to the study of the physical, chemical, and thermodynamic properties of hydrazine and some of its derivatives.

2. From an analysis of the information available from past and present research on hydrazine and its derivatives, the conclusion was drawn that solutions of one or more of the five methylhydrazines in hydrazine

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should constitute a superior rocket fuel. A calculation of the approximate performance of methylhydrazine at a single mixture ratio showed that a slight increase in specific impulse over that of hydrazine was possible. Calculations have not yet been made for the more highly methylated hydrazines, but it is believed that their performance will not be greatly different from that of hydrazine itself. A study of the chemical and physical properties of the methylhydrazines has been instituted as a consequence of these considerations. Since it is necessary to prepare the substituted hydrazines for use in these studies, much information is currently being obtained on methods of preparation and purification of the methylhydrazines.

## B. METHYLHYDRAZINE

1. A sample of anhydrous methylhydrazine was necessary for the determination of the freezing-point composition curve of the system hydrazine-methylhydrazine. The preparation of such a sample was initially believed to be no problem. However, the method of isolating the anhydrous base by neutralization of an acid salt in concentrated aqueous solution, although applied with success in this laboratory to hydrazine, was soon proved inadequate for methylhydrazine.

2. A large quantity (130 g) of methylhydrazine sulfate therefore was prepared by the method of Katt.\* Several methods were then tried in an effort to liberate the anhydrous base from the sulfate. The first method employed reportedly gives excellent results,\*\* but when attempted in this laboratory, an aqueous solution containing only 17% methylhydrazine was obtained, although the recovery of free base was virtually 100%. The referenced procedure requires that potassium hydroxide pellets be added to the solid methylhydrazine sulfate, the mass heated, and water then slowly added through a dropping funnel. The distillate obtained by this procedure boiled at 104°C, 17° higher than methylhydrazine, and was obviously a water-methylhydrazine azeotrope.

3. The preparation of anhydrous hydrazine by a method that does not require the addition of water\*\*\* was next attempted. This procedure involves the low-temperature fusion of the acid salt with an excess of fused potassium hydroxide, and subsequent low-pressure distillation of the free base. Much higher temperatures were required with the methylhydrazine sulfate than are necessary with hydrazine hydrochloride. Although the bath temperature was 70°C above the melting point of the sulfate, a yield of only 40% free base resulted from the fusion, and the distillate was only 50%

---

\* Organic Synthesis, Coll. Vol. 2, John Wiley & Sons, New York, 1943, p. 395.

\*\* R. D. Brown, et al., "Studies in the Preparation, Analysis, and Thermal Decomposition of Hydrazine, Methyl and Ethyl Hydrazine," Project No. 349, University of Alabama, 1 November 1948 (Unclassified).

\*\*\* E. L. Bulgozely and E. L. Wanger, J. Am. Chem. Soc. 73, 5866 (1951).

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II Study of Hydrazine Derivatives, B (cont.)

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methylhydrazine. However, during these experiments, the discovery was made that the addition of a small quantity of potassium hydroxide to the liquid caused an immediate separation into two layers, the upper of which contained 73% methylhydrazine as determined by acid titration.\* Further addition of a quantity of fused hydroxide, and distillation of the decanted liquid gave a product of 96% purity. Penneman and Audrieth\*\* have observed a similar separation of two layers in mixtures of sodium hydroxide, hydrazine, and water above 60°C, but not with potassium hydroxide nor at as low temperatures as were observed in these experiments. The commercial application of such enrichment methods is made attractive by the fact that a simple distillation of the upper phase yields a material sufficiently anhydrous to use directly.

4. Since adequate heat transfer through the semi-solid reaction mass was not obtained in the dry distillation method outlined above, the addition of a high-boiling alcohol to aid in the achievement of better heat transfer, with resulting higher yields and purity of the product, was tested. Methylhydrazine sulfate and fused potassium hydroxide were heated under reflux conditions in the presence of isoamyl alcohol, followed by a crude fractionation of the supernatant liquid. An 85% recovery of free base of 87% purity was achieved; a high-efficiency spinning-band distillation column will be used in an attempt to increase the purity of the methylhydrazine.

## C. METHYLATION OF HYDRAZINE

1. Harries and Haga,\*\*\* and Westphal\*\*\*\* have investigated the formation of alkylhydrazines by the action of alkyl halides on hydrazine. An extension of this work is now in progress, in which an attempt is being made to determine whether a low-freezing solution of various methylhydrazines in hydrazine can be conveniently prepared by direct alkylation of hydrazine. As only two reactions, one using methyl iodide and the other methyl bromide, have thus far been conducted, it is not yet possible to comment on the feasibility of this method, although it is hoped that ultimately it may be developed into a successful preparative process.

2. When 0.28 mole of methyl iodide was slowly added to 0.28 mole of hydrazine (as a 50 wt% solution in ethanol) at room temperature, a vigorous reaction occurred, and a white solid formed. After heating this slurry for 3.5 hr at 50°C, potassium hydroxide was added and the mixture was again heated briefly in an attempt to decompose alkylhydrazine salts formed by reactions such as  $N_2H_4 + CH_3I \longrightarrow CH_3HNHNH_2 \cdot HI$ . The reaction mixture was

---

\* Work is now in progress to determine the effect of temperature on this ternary system and the amount of hydroxide in the upper layer.

\*\* J. Am. Chem. Soc. 71, 1644-1647 (1949).

\*\*\* Ber. 31, 56-64 (1889).

\*\*\*\* Ber. 74B, 759 (1941).

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then extracted with ether (in which methylhydrazines, but not hydrazine are soluble), and this solution was analyzed for nitrogenous substances. Since only a trace of nitrogen was found, and since more methyl iodide was not immediately available, a more extensive investigation using the less expensive methyl bromide seemed advisable, rather than further study of the solid formed in this reaction.

3. The reaction of equimolar amounts was again carried out, 87 g (0.92 mole) of methyl bromide being used; a white solid immediately formed as the halide contacted the hydrazine. All of the alcohol was removed by vacuum distillation; isolation of basic materials was attempted by the method described in Paragraph II, B, 2. The 33 g of liquid obtained contained a total of 0.34 mole of monoacidic bases as determined by titration with hydrochloric acid. When further treated with potassium hydroxide and distilled, this solution yielded fractions boiling at 45 to 50°, 84 to 92°, and 110 to 114°C. The highest-boiling fraction is undoubtedly hydrazine, but the lower-boiling ranges do not correspond to any of the ~~the~~ methylhydrazines. Molecular weight determinations will be performed in an effort to rapidly identify the materials. The apparatus of Nash\* has been constructed for this purpose. Although this equipment has not yet been used to determine the molecular weight of the low-boiling fractions, check runs made with hydrazine prove the method to be accurate within 1%. The solid mass from which the liquid fractions were distilled was refluxed with isocamyl alcohol and potassium hydroxide, the liquid was decanted from the solid, and then distilled. Complete results are not yet available, but from the large amount of material boiling at 114°C (i.e., hydrazine), it appears that the course of the reaction results in the production of trimethyl azonium bromide,  $[(CH_3)_3NNH_2] Br$ , and hydrazine hydrobromide. In the belief that the reaction can be stopped before the azonium salt forms, preparations in which the hydrazine is used in lower concentration will be attempted. Addition of methyl bromide to a refluxing mixture of potassium hydroxide and hydrazine is also being considered as a one-step method for the preparation of substituted hydrazines.

4. In the consideration of methylating agents, the use of dimethyl sulfate is an obvious one. That it has not been employed to any great extent in direct reaction with hydrazine is perhaps due to the fact that a series of compounds can result, hence the reaction is inefficient when one specific product is desired. Since a simple experiment verified the presence of methylhydrazine (isolated as the picrate) in a small quantity of hydrazine to which dimethyl sulfate had been added, the reaction is now being conducted on a larger scale to determine whether methylhydrazines form in sufficient yields to justify further study of the method, or whether methylation proceeds to the azonium salt.

5. Although even more precautions were observed in handling solutions of methylhydrazine than have been used in the past with hydrazine, laboratory personnel have repeatedly suffered from headaches after working

\* Anal. Chem. 23, 1868-70 (1951).

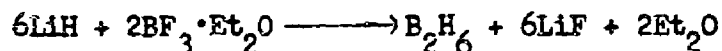
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with this compound for short periods of time. Until conclusive evidence is found indicating that methylhydrazine is at least no more toxic than hydrazine, rigorous safety precautions will be observed at all times during this investigation.

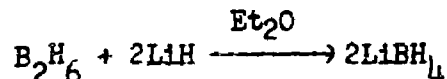
## III. PILOT-PLANT PREPARATION OF LITHIUM BOROHYDRIDE IN HYDRAZINE

A. Pilot-plant production of a 13% solution of lithium borohydride in hydrazine has been initiated. Details of this program, the objective of which is to produce a sufficient quantity of the low-freezing solution for evaluation in a 100-lb-thrust rocket motor, have been described before.\* Briefly, the four steps of the process are:

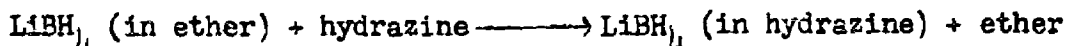
1. Generation of diborane from lithium hydride and boron fluoride:



2. Absorption of diborane by lithium hydride to give lithium borohydride:



3. Formation of a concentrated solution of lithium borohydride in hydrazine by addition of the lithium borohydride-ether solution to hydrazine, followed by evaporation of the ether:



4. Dilution of the solution to 13.2% lithium borohydride.

B. One pilot-plant run has been completed through Step 3, and three additional runs are in process in Step 3. During the first trial run, only a low yield of diborane was produced in Step 1, hence very little lithium borohydride formed in Step 2. As it was known that the use of methyl borate often increases the yield of diborane when made by the method in Step 1, 8 mole% of this material was added in the next three runs; results observed in these runs favor the continued use of methyl borate.

C. Attempts to remove the ether in Step 3 revealed that a higher temperature (60°C) was required than laboratory work had shown to be necessary. This was noted when a blend of the materials from two runs was found to consist of a slurry composed of white solids, hydrazine, and considerable amounts of ether, even though the solution had been heated for some time at 40°C. When a sample of this material was removed from the metal apparatus and heated to 60°C, a clear solution, containing only a small quantity of an

\* Aerojet Report No. 577, 7 February 1952 (Confidential).

easily filterable flocculent precipitate, resulted. Thus it appears that complete removal of the ether through heating to a higher temperature is a necessary step in dissolving lithium borohydride in hydrazine.

D. A tabulation of the results of all runs conducted to date, together with conditions of time and temperature, are shown in Table I. A view of the production apparatus is shown as Figure 1.

#### IV. KINETIC STUDY OF THE THERMAL DECOMPOSITION OF NITROMETHANE

##### A. INTRODUCTION

It is believed that a more adequate understanding of the chemical processes occurring during the thermal decomposition of nitromethane would be of considerable assistance in the solution of problems related to the use of nitromethane as a rocket monopropellant. An intimate understanding of such chemical processes can only be gained through extensive studies of reaction rates, which lead eventually to the establishment of the reaction mechanism. It is characteristic of vapor-phase decomposition reactions that mechanisms can be determined only by a great number of rate experiments. Such is the case with nitromethane; all studies to date have failed to resolve explicitly the mechanism of the reaction. Despite the present lack of success in elucidating the mechanism, the problem is by no means considered unsolvable. The kinetic experiments conducted by the various observers have established the rate of decomposition for one set of conditions and have allowed determinations of the activation energy of the decomposition reaction. It is believed that if the results obtained thus far can be extended to include rate experiments under widely varied conditions of pressure and concentration of reaction products, then the proper rate law and reaction mechanism can be developed. Toward that end, work is now in progress on the kinetics of nitromethane decomposition at pressures of 13.5 atmospheres, amounting to nearly a 30-fold increase in pressure over the earlier experimental conditions.

##### B. PRELIMINARY TESTS

1. Exploratory experiments showed that 3-ml ampoules made of standard-wall Pyrex tubing are capable of withstanding pressures of 1000 psi, and that the thermal shock encountered upon "quenching" the reaction by plunging the ampoules into water would not break them. Having established the fact that glass could be used in this work it was then necessary to determine a temperature range in which kinetic studies could be carried out conveniently. To do this, ampoules were prepared which contained sufficient nitromethane to develop pressures of approximately 400 psi at the temperatures selected. These samples were then placed in a heated fused-salt bath for half-hour intervals. At the end of the heating period, the ampoules were rapidly cooled and their contents were analyzed in a mass spectrometer.

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## IV Kinetic Study of the Thermal Decomposition of Nitromethane, B (cont.)

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2. The relative extent of decomposition was determined by comparison of the peak height at mass 61 (nitromethane) with those at masses 44 and 18, the parent peaks of carbon dioxide and water. The following qualitative observations were made:

Temp., °C	210	310	360
Decomposition	Slight	Appreciable	Extensive

As these experiments were conducted only to determine the temperature range at which nitromethane decomposes at high pressure, no efforts were made to obtain quantitative kinetic data. On the basis of these observations it has been decided to perform the first accurate decomposition studies in the temperature range of 300 to 320°C. This temperature range is approximately 100°C lower than that found suitable for previous studies at subatmospheric pressure.\*

### C. DESIGN OF APPARATUS

The apparatus employed for the preliminary decomposition studies could not be used for this investigation since it permitted only one sample to be tested at a time, and did not provide for precise temperature control. In order to overcome these difficulties a more refined apparatus was designed. It consists of the following principal components: a furnace unit, a Leeds and Northrup Electromax controller, a safety shield, and an assembly for the introduction and removal of samples by remote control. The furnace is the same as that used in an earlier study of nitromethane\*\* but it has been modified in order to permit the use of "Blue Temp" ( $\text{NaNO}_2\text{-NaNO}_3$  mixture) as the heating bath, and to allow simultaneous heating of four ampoules. The remote-control assembly permits the operator to introduce and remove ampoules by the use of a hydraulically operated arm controlled through appropriate slots in a protective box constructed of 1/2-in. steel plate. All components are ready for use except the remote-control assembly which is being constructed at present. Diagrams of the equipment will be included in the next report.

### D. PURIFICATION OF NITROMETHANE

1. The supply of nitromethane at hand was obtained three years ago from the Commercial Solvents Corporation and was purified at that time by steam and vacuum distillations in this laboratory. The purified material was placed in Pyrex bottles and stored in the dark. As a recent water analysis by the Karl Fischer method showed that it contained 0.1% water, and since the purification had been carried out three years ago, it was necessary to redistill a portion of the nitromethane. The distillation was accomplished in an

\* Aerojet Report No. 493, 1 February 1951 (Confidential).

\*\* Ibid.

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## IV Kinetic Study of the Thermal Decomposition of Nitromethane, D (cont.)

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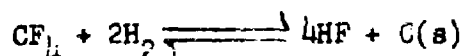
Oldershaw column at a reflux ratio of 5 to 1. Nitromethane (bp 101.2°C) and water form an azeotrope at 83.7°C, containing 21.8 wt% water, and therefore the first fraction distilling over should be rich in water. This was found to be the case, the first fraction having a water content of 1.6%. In subsequent fractions the water content dropped to less than 0.05%. The fraction selected for use in the decomposition experiments has a water content of 0.04%, is colorless in appearance, has an infrared spectrum corresponding to that reported by Smith,\* and has a freezing point of -29.1°C (Figure 2).

2. The apparatus used for the determination of the freezing point is that of Herington and Handley,\*\* and was previously employed for the determination of the freezing point--composition curve for the hydrazine-unsym.-dimethylhydrazine system; it is shown in Figure 3. It offers the advantages of requiring a sample of less than 2 ml, absence of any metal components, and ease of handling of hygroscopic materials. A sample is introduced with a hypodermic syringe through a vaccine stopper into the glass U-tube. A test tube is placed about the U to achieve air insulation, and a Dewar flask containing a bath approximately 20 degrees below the expected freezing temperature is then placed around this tube. The mercury pressure-interrupter causes a fluctuation (100 cycles/min) in the blanketing nitrogen pressure, thus continually oscillating the cooling sample over glass projections within the U-tube and effectively stirring it. Temperature is recorded by a Kel-F-coated copper-Constantan thermocouple.

## V. ENGINE TEST PROGRAM

### A. PRELIMINARY TESTING OF AVIATION GASOLINE WITH OXYGEN AND FLUORINE MIXED OXIDIZER

1. The high cost and short supply of elemental fluorine detract greatly from its acknowledged superiority as an oxidizer in systems in which it is necessary to obtain the greatest possible combustion energy. In addition, fluorine is limited in applicability to use with noncarbonaceous fuels, since it has been observed experimentally at Aerojet, and calculations verify the observation,\*\*\* that on combustion of carbon-containing fuels under fuel-rich conditions, a heavy deposition of free carbon occurs. That is, the equilibrium



lies far to the right at high temperatures. The possibility that an oxidizer consisting of a mixture of fluorine and oxygen might overcome both of the

\* D. C. Smith, C. Y. Par, and J. R. Nielson, J. Chem. Phys. **18**, 706 (1950).

\*\* J. Chem. Soc. 199 (1950).

\*\*\* Aerojet Report No. 577, 7 February 1952 (Confidential).



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V Engine Test Program, A (cont.)

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above objections to fluorine prompted a short series of tests in the small test engine of the combination aviation gasoline—liquid fluorine—liquid oxygen. It was believed that by adding the proper amount of oxygen, combustion efficiency would be improved, inasmuch as the carbon normally resulting from combustion with fluorine would be converted completely to carbon monoxide and carbon dioxide.

2. Although funds allotted to the present contract were not used to support the tests, the small fluorine-test engine which is under the jurisdiction of the group associated with this contract was employed; for this reason, and because this type of information is particularly pertinent to the present program, the results obtained during the series of 8 tests are reported here. It is realized that the eight tests performed cannot be considered conclusive, since it would be necessary to vary the ratio of oxygen to fluorine as well as the over-all total oxidizer-to-fuel ratio, in order to determine the exact optimum conditions. The only intended purpose of the program was to determine whether a mixture of fluorine and oxygen showed promise as a high-performance oxidizer for hydrocarbons.

3. A sample of aviation gasoline with the following properties was used:

Aromatics, %	3.4
Bromine No.	1
Reid Vapor Pressure, psi	6.71
Specific Gravity	0.694

Fluorine was obtained from the Pennsylvania Salt Manufacturing Co.; the test facilities were those previously described in the testing of dimethylaminodiborane with fluorine.\* Gaseous oxygen and fluorine were mixed by pressurizing oxygen into a partially filled fluorine cylinder, the increase in pressure being used to calculate the ratio of fluorine to oxygen; at least two hours was allowed before liquefaction of the mixed gases was accomplished by cooling with liquid nitrogen.

4. A total of 8 tests were made. The one test with 100% oxygen as the oxidizer was made in order to have a point of comparison for the particular motor configuration used. The following were the maximum specific impulse and characteristic exhaust-velocity values obtained:

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\* Aerojet Report No. 527, 31 July 1951 (Confidential)

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V Engine Test Program, A (cont.)

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<u>Specific Impulse</u> <u>lb-sec/lb</u>	<u>Characteristic</u> <u>Exhaust Velocity</u> <u>ft/sec</u>	<u>Mixture</u> <u>Ratio,</u> <u><math>\frac{w_{ox}}{w_f}</math></u>	<u>Oxidizer,</u> <u>mole%</u> <u>Fluorine</u>
261	5684	4.62	50.3
262	5990	4.62	66.7
(206)	(5016)	(2.51)	(100% oxygen)

5. A complete listing of the test results is presented in Table II. The incorporation of 50 mole% fluorine in the oxidizer increases the performance markedly over that of pure oxygen. However, further increase in the fluorine content of the oxidizer to 67 mole% appeared to effect no further significant increase, although the tests with this oxidizer composition are not numerous enough to dispel all uncertainty. Figure 4 shows performance values plotted against over-all mixture ratio; specific impulse is presented as a function of molar mixture ratio in Figure 5.

## B. MODIFICATIONS IN THE TEST ENGINE

1. The thrust of the test engine that is used for the evaluation of propellants under this contract has now been increased from 50 lb to 100 lb. The thrust chamber now has the following dimensions:

Characteristic length of combustion chamber, $L^*$	128 in.
Diameter of nozzle throat section, $D_t$	0.558 in.
Exit to throat area ratio, $A_e/A_t$	4.42

2. The minor expense of modifying the tester was more than justified by the following advantages:

a. The heat-loss correction has been reduced. Since heat transfer per unit time still remains almost the same, and the amount of propellant burned is now doubled, this factor is now much less than when the 50-lb thrust engine was used. A stainless steel liner has replaced the copper liner, thus reducing the heat-transfer correction even further.

b. Errors in measurement of the throat area, caused by solid deposition, should be reduced. This improvement in accuracy results from the fact that, although solid deposition may still occur, the depth of the deposits should remain the same, but the throat area has been doubled.

c. The net consumption of fuel during a test will be only slightly more than when the 50-lb motor was used, as preliminary tests indicate that the time required to achieve steady-state conditions is now 60% of the former value.

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V Engine Test Program (cont.)

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## C. HYDRAZINE-OXYGEN CALIBRATION TESTS

1. After modification of the test engine it was necessary to conduct a number of firings to ensure proper functioning of the unit. As a base curve for the comparison of the 13% solution of lithium borohydride in hydrazine was desired, the checkout tests were made with the hydrazine and oxygen system.

2. A 1:1 impinging injector with 10 paired holes was designed for use with hydrazine (Figure 6). A 70°-impingement angle is used; resultant momentum is axial at a mixture ratio of 0.84, and the point of impingement is 1/16 in. from the face of the injector.

3. Ten tests were made with 95% hydrazine without incident. The new injector design and motor configuration performed very satisfactorily. A maximum specific impulse corresponding to 93% of theoretical was obtained. Heat-transfer corrections ranged from 1.62 to 3.60%, many times lower than the factors previously encountered. Performance is shown as a function of mixture ratio in Figure 7. A summary of performance data is given in Table III.

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TABLE I

## PILOT-PLANT PREPARATION OF LITHIUM BOROHYDRIDE IN HYDRAZINE

<u>Step 1</u>									
Run No.	<u>LiH</u>		Ether Liters	<u>B(OCH<sub>3</sub>)<sub>3</sub></u>		<u>BF<sub>3</sub>·Et<sub>2</sub>O</u>		Temp. °C	Time hr
	<u>g</u>	<u>mole</u>		<u>g</u>	<u>mole</u>	<u>g</u>	<u>mole</u>		
1	207	26.1	3.28	—	—	2120	14.9	30-35	3.25
2	267.5	33.6	1.75	289	2.78	2650	18.65	32-40	1.83
3	267.5	33.6	1.75	289	2.78	2650	18.65	25-35	1.75
4	446	56.6	2.91	479	4.62	4400	31.0	35-40	4.00

<u>Step 2</u>								
Run No.	<u>LiH</u>		<u>Ether Liters</u>	<u>Temp. °C</u>	<u>Time hr</u>	<u>Purity LiBH<sub>4</sub> %</u>	<u>Weight LiBH<sub>4</sub> g</u>	<u>Yield %</u>
	<u>g</u>	<u>mole</u>						
1	39	4.92	4.25	22-24	3.25	38.2	—	—
2	78.5	9.89	6.0	24-28	1.83	99.4	187.5	58.4 <sup>1</sup>
3	78.5	9.89	6.0	25-35	1.75	83.8	85.5	39.9
4	131	16.5	10.0	22-25	4.00	94.5	235.5	66.3

<sup>1</sup> Based on LiH used in Step 2 of Runs 1 and 2.

Table I

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TABLE II

## AVIATION GASOLINE AND MIXTURES OF LIQUID FLUORINE AND LIQUID OXYGEN

## SUMMARY OF PERFORMANCE DATA

Test No. PP-15-1-	Fuel	Oxidizer				Chamber Pressure psia	M.R. ( $w_{ox}/w_f$ )	$C_F$ (Corr. for Heat-Trans.)	$I_{sp}$ (Uncorr.) lb-sec/lb	$c^*$ , ft/sec (Corr. for Heat-Trans.)	$I_{sp}$ , lb-sec/lb, (Corr. for Heat-Trans.)	Molar Mixture Ratio
		Mole%		Wt%								
		F <sub>2</sub>	O <sub>2</sub>	F <sub>2</sub>	O <sub>2</sub>							
1	Aviation Gasoline	50.7	49.3	55.0	45.0	225	2.57	1.34	192	4708	196	8.29
2		50.7	49.3	55.0	45.0	330	3.82	1.46	244	5541	252	12.32
3		—	100	—	100	241	2.51	1.32	198	5016	206	8.86
4		50.3	49.7	54.6	45.4	319	5.85	1.43	242	5665	252	18.88
5		50.3	49.7	54.6	45.4	322	4.62	1.43	253	5884	261	14.91
6		50.3	49.7	54.6	45.4	316	4.87	1.43	243	5736	255	15.72
7		66.7	33.3	70.4	29.6	321	4.62	1.41	247	5991	262	14.50
8		66.7	33.3	70.4	29.6	305	3.87	1.42	237	5648	249	12.15

Table II

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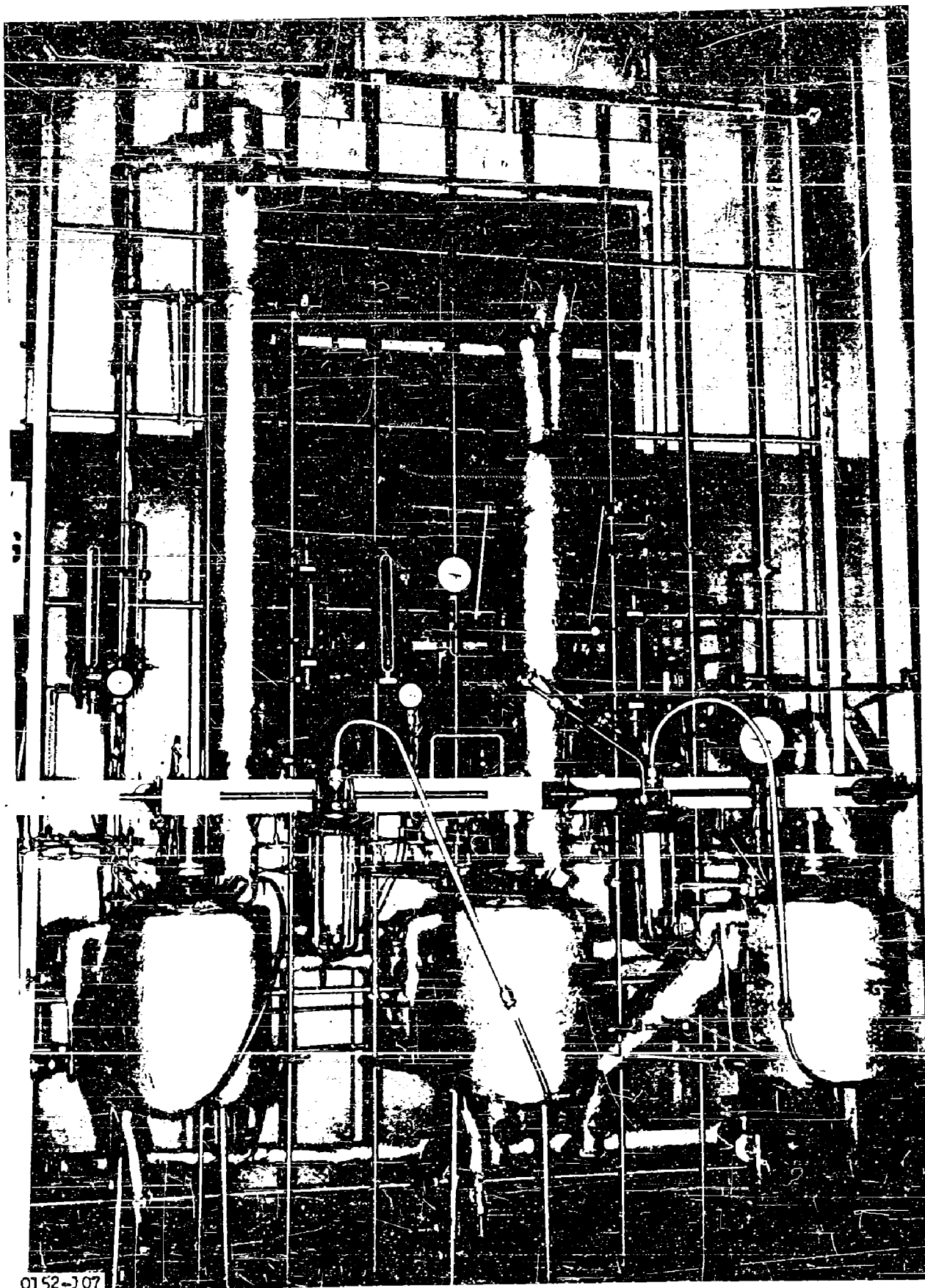
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TABLE III  
HYDRAZINE AND LIQUID OXYGEN  
SUMMARY OF PERFORMANCE DATA

Test No. 37LF-	Fuel	Oxidizer	Chamber Pressure, psia	M.R. ( $w_{ox}/w_f$ )	$C_F$ (Corr. for Heat-Trans.)	$I_{sp}$ (Uncorr.) lb-sec/lb	$c$ , ft/sec (Corr. for Heat-Trans.)	$I_{sp}$ , lb-sec/lb, (Corr. for Heat-Trans.)	Remarks
1	↑	↑	320	1.20	1.39	243	5737	247	
2	↑	↑	304	0.96	1.37	239	5758	245	
3	↑	↑	289	0.70	1.37	242	5868	249	
4	↑	↑	306	1.01	1.38	234	5598	239	Injector temp. recorder did not work. Heat-trans. to injector is minor and was neglected.
5	Hydrazine	Liquid Oxygen	289	0.85	—	241	—	—	Chamber coolant flow and injector temp. did not record.
6	↓	↓	289	0.83	1.37	238	5809	247	
7	↓	↓	298	0.98	1.38	242	5835	251	
8	↓	↓	291	0.71	1.39	241	5872	254	
9	↓	↓	291	0.83	1.41	244	5839	255	
10	↓	↓	298	0.83	1.38	244	5850	250	

Table III

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Pilot Plant for Preparation of Lithium  
Borohydride-Hydrazine Solution

Figure 1

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COOLING CURVE

NITROMETHANE

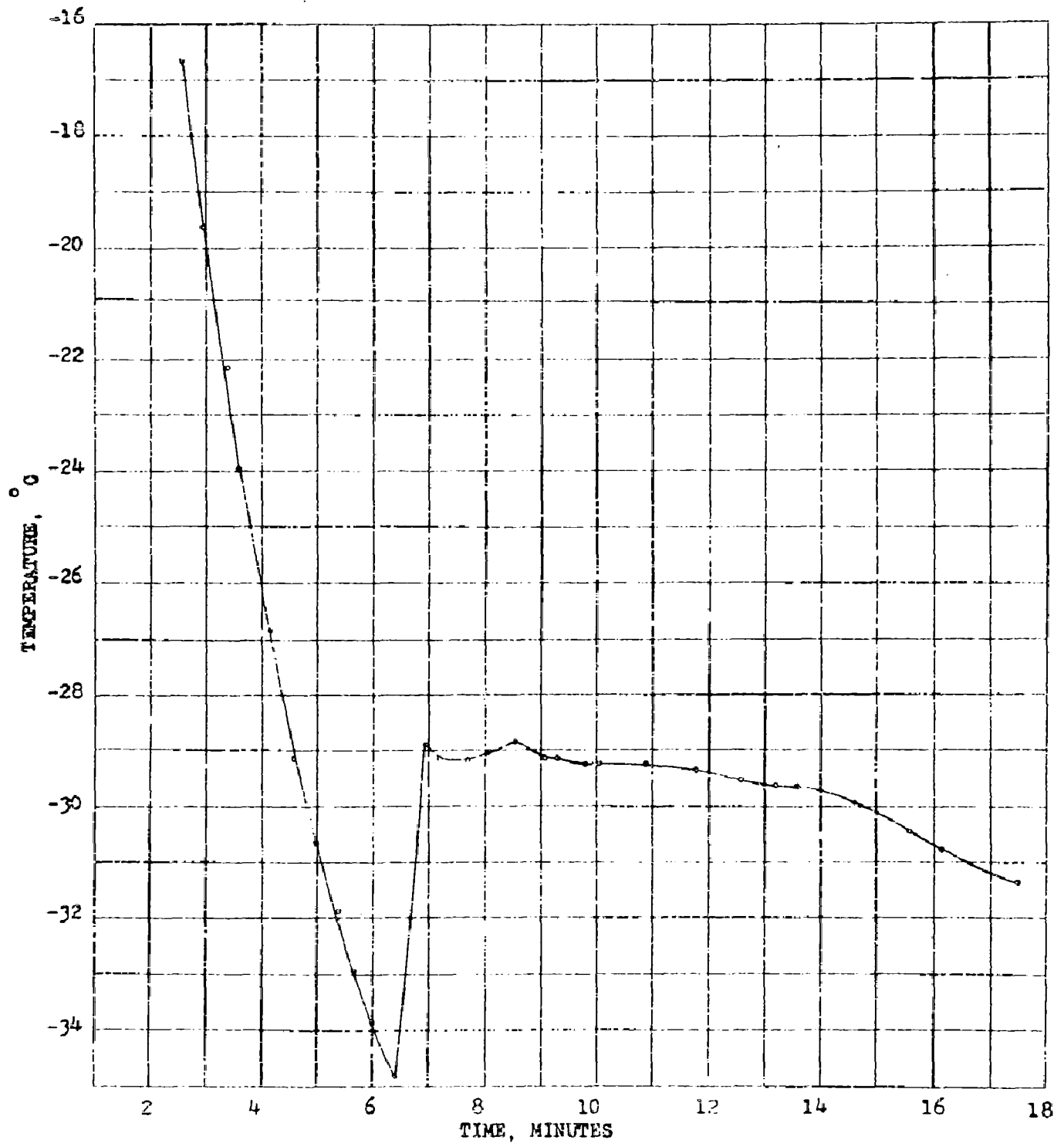


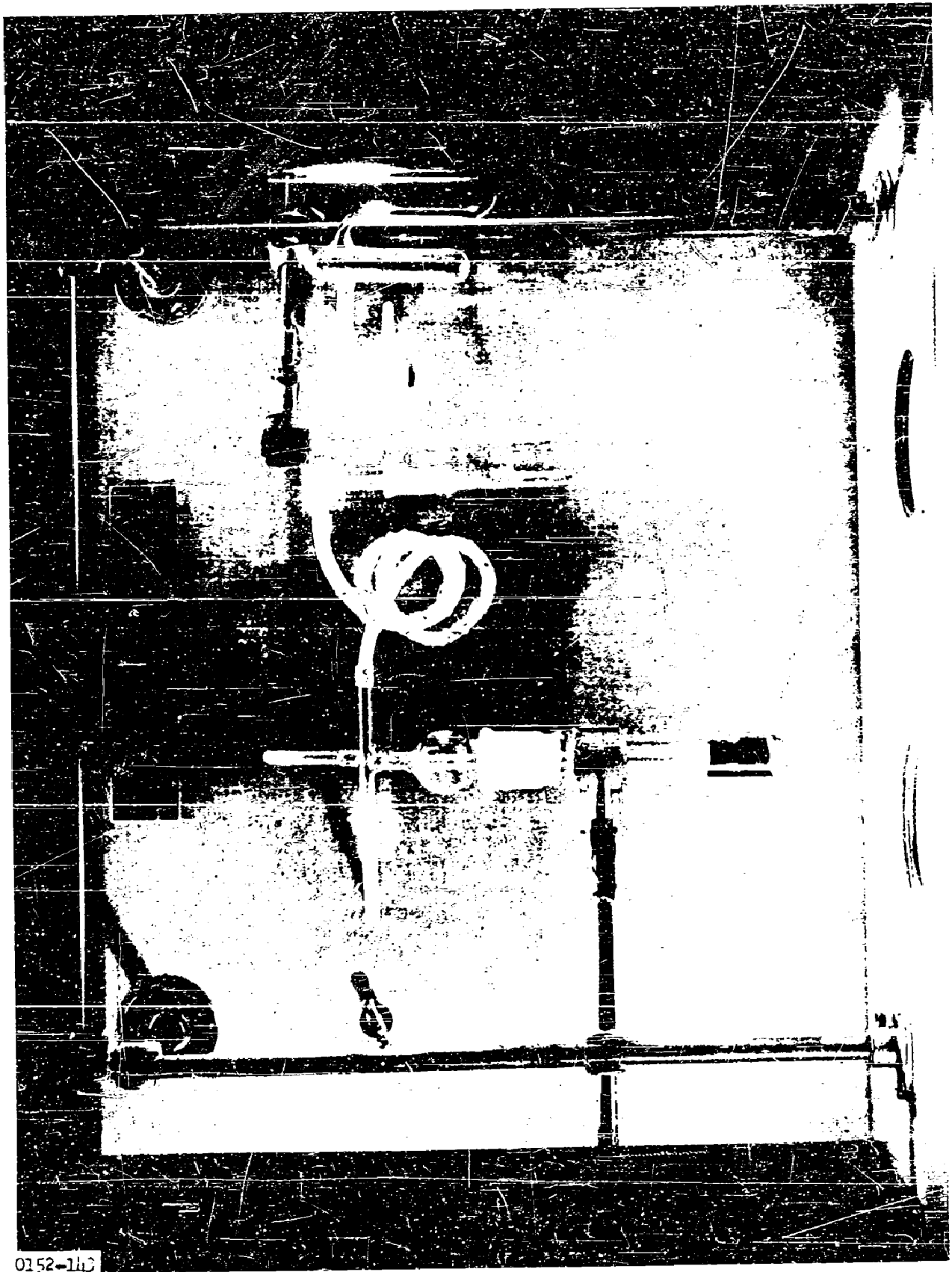
Figure 2

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Figure 3

PERFORMANCE VERSUS MIXTURE RATIO  
FOR AVIATION GASOLINE AND MIXTURES OF  
OXYGEN AND FLUORINE

$P \approx 50$  lb  $P_c \approx 300$  psia  
 100%  $O_2$  50 Mole %  $F_2$  66.7 Mole %  $F_2$

Tests PP15-1. 1 through 8

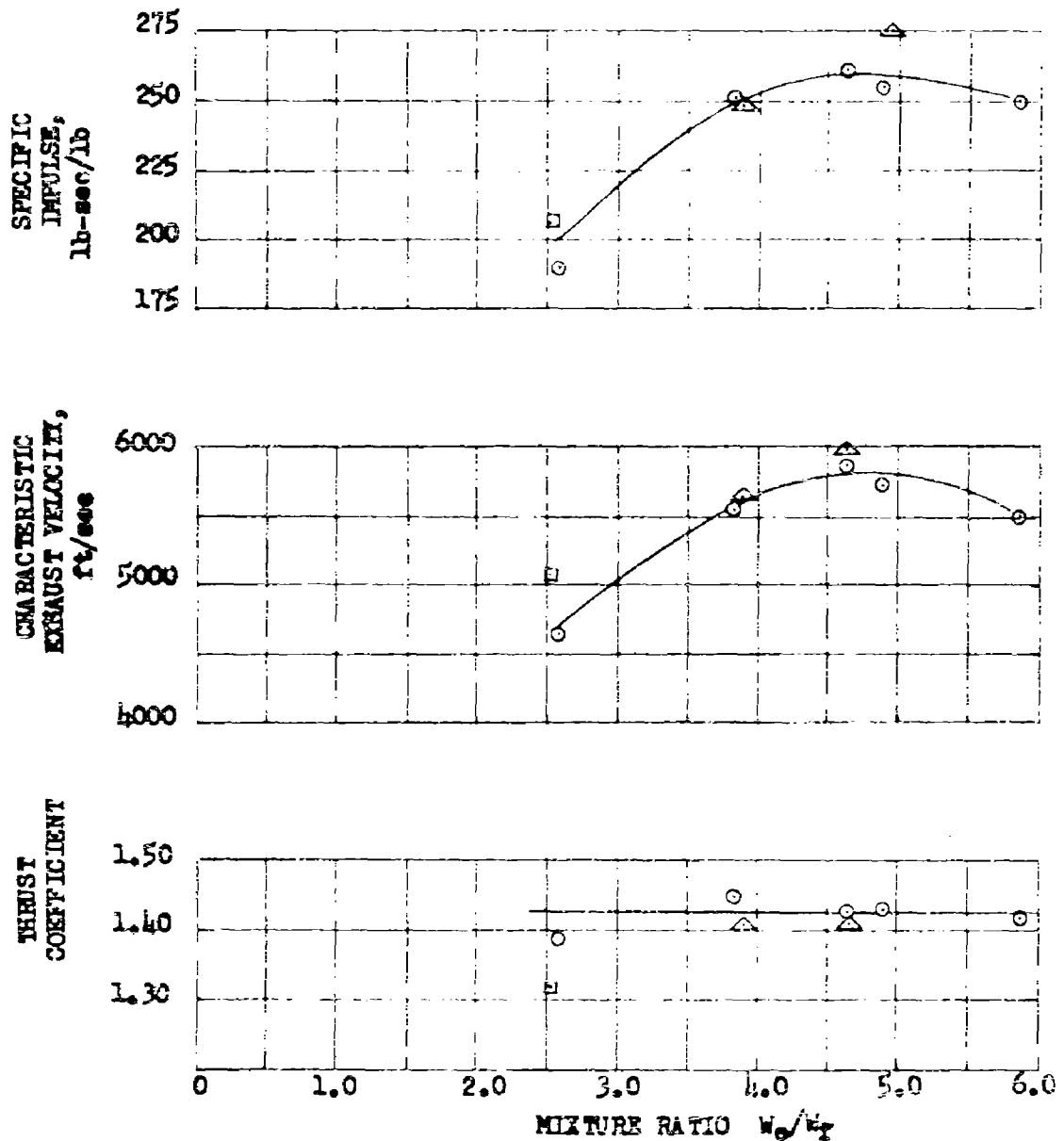


Figure 4

U-3184 CLR/CSE 2-29-52

SPECIFIC IMPULSE,  $I_{sp}$ , lb-sec/lb

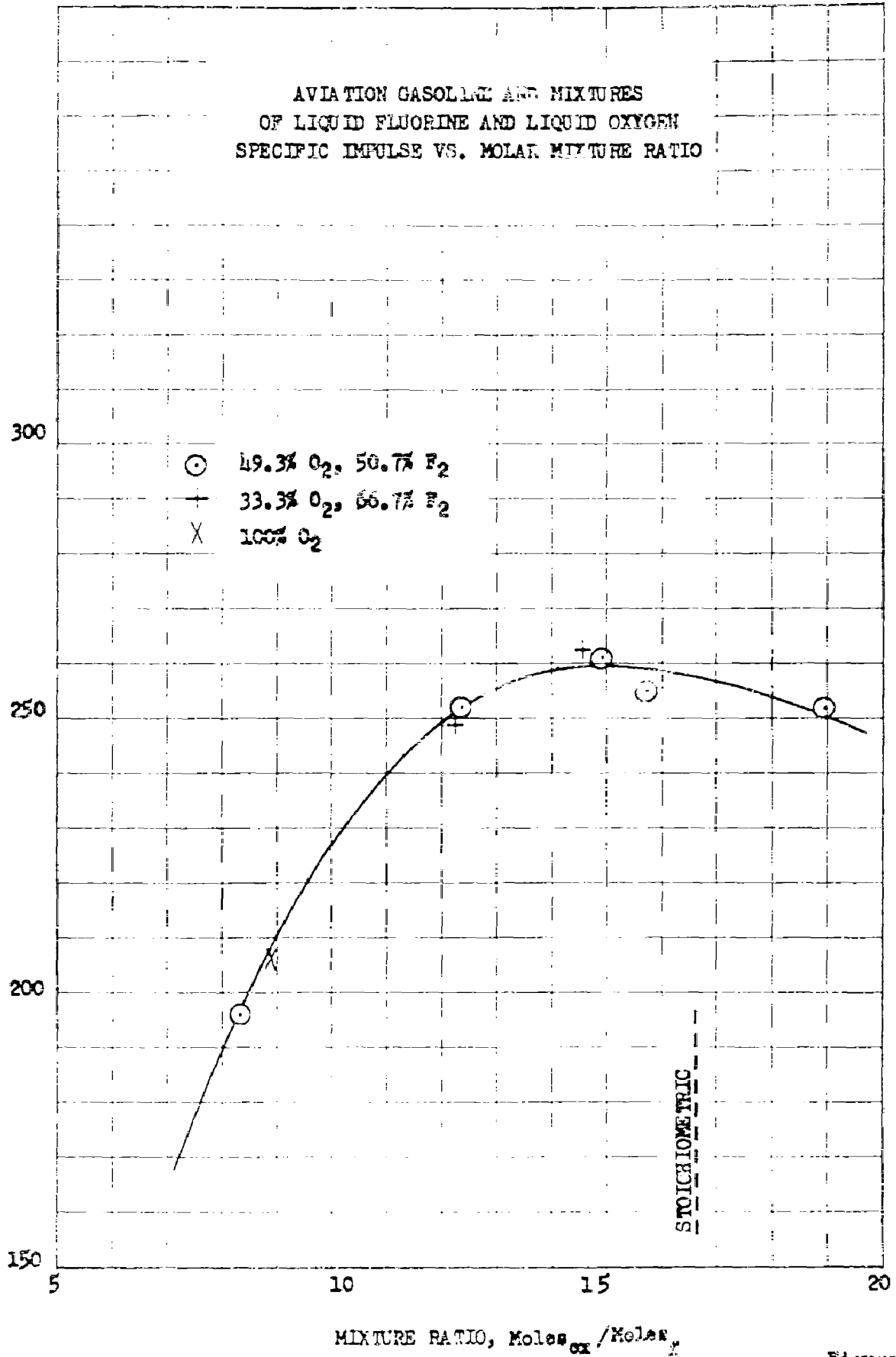


Figure 5



NOTE: THIS VIEW FOR REWORK OF ②

ASSEMBLY

[illegible]

PERFORMANCE  
VERSUS  
MIXTURE RATIO  
FOR HYDRAZINE AND LIQUID OXYGEN

$F = 100 \text{ lb}$       • With Turbulator  
 $P_o \approx 300 \text{ psia}$   
Test Nos. D37LF 1-4, 6-10 • Without Turbulator

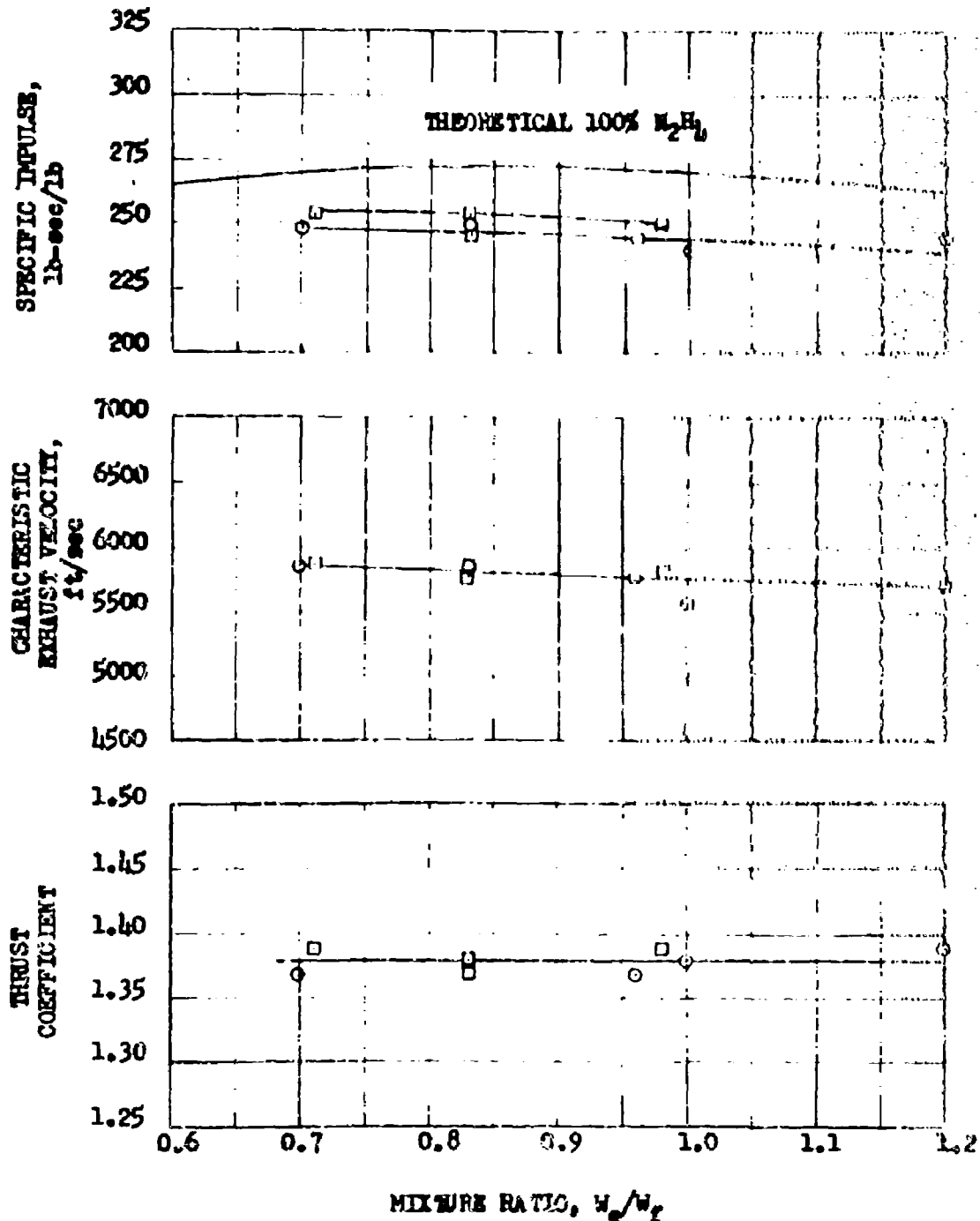


Figure 7

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